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An Investigation of
Fluid Extrusion of Metals
October 1, 1963 - January 1, 1964

Contract NASw-742

to

National Aeronautics and Space Administration
Washington, D.C.

by

Pressure Technology Corporation of America
433 Amboy Avenue
Woodbridge, New Jersey

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ABSTRACT

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Deformation-under-pressure tests have been conducted on NASA Nickel Base TAZ-8, Series 300 Mar-Aging Steels, Magnesium Oxide, Tungsten-4.5% Tantalum Alloy, and Chrome-Magnesia Composite. The results are given as well as some interpretation of them.

Author

INTRODUCTION

The previous progress report had detailed the fluid-to-fluid extrusion of both annealed and hardened 300-series maraging steel. It is of interest that the hardened maraging steel probably constitutes the highest strength material that has been extruded to date in the world. The question consequently arises as to whether an upper limit exists for strength of billet material that is to be fluid-to-fluid extruded. A general answer to this question can be given by noting the analogy between wire drawing and fluid extrusion. A comparison exists in reference 1, page 42, among ram extrusion, fluid extrusion and wire drawing. It is seen from this reference that in many ways fluid-to-fluid extrusion is closer to drawing than extrusion. Experimental evidence on the analogy between fluid-to-fluid extrusion and wire drawing is presented in reference 2 where a billet oversized for the entrance to a fluid-to-fluid-extrusion die was found to be drawn in radially towards the center line of the die before actually touching the die, just as exists in wire drawing practice. If a close analogy is thus assumed to exist between wire drawing and fluid-to-fluid extrusion, one might expect the same

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lack of limitation to apply to fluid-to-fluid extrusion as to wire drawing, namely, that the hardest metals can be successfully reduced by passage through a die without fracture of the die. It might consequently be concluded that there will be no limit to the strength or hardness of billet material that can be passed through a fluid-to-fluid-extrusion die without fracture of the die.

The work during this period comprised of extension of the fluid-to-fluid extrusion and tensile tests under pressure to other materials and other sets of test conditions.

This work was conducted under NASA contract NASw-742.

EXPERIMENTAL

Chrome-30 (Chromium-MgO Composite) - Pressurization

One of the test materials in this program is a dispersion of MgO in chromium metal, termed Chrome-30 by The Bendix Corporation which manufactures it. This material represented the class of dispersion-hardened mixtures. Recently, however, experimental results from Australia (reference 3) on the exposure of chromium to hydrostatic pressure heightened interest in Chrome 30.

The per cent reduction in area for Chrome-30 averages between 13 and 16 per cent at room temperature according to personal communication with a representative of The Bendix Corporation. The 0.2% yield strength simuli is given as about 30,000 psi to 30,200 psi, and UTS about 46,000 to 48,000 psi. Per cent elongations are similarly given as 20 to 25 per cent in tensile tests.

The Australian data indicate that recrystallized chromium which possessed 0% elongation and 0% reduction in area became ductile after having been subjected to fluid pressure of 10,000 atmospheres (approximately 150,000 psi). After this pressurization treatment, the per cent elongation was 65.4 and the per cent reduction

in area was 47.3. Repeated pressurization (five applications to the same pressure) produced the same results as the single pressurization. The tensile specimens possessed 1" gage length and were 0.14" in diameter, finished by electro polishing. Nitrogen content was .0008 - .005%, with the results given pertaining to the higher nitrogen contents. Chrome of the lowest nitrogen content remained brittle after the pressurization treatment. The pressurization treatment also tended to eliminate the upper yield point.

It may be noted that personal communication from Dr. A. Gilbert, Metal Science Group, Battelle Memorial Institute, cites efforts by B.A. Wilcox, A.R. Rosenfield, and G.T. Hahn to duplicate this use of pressure to obtain ductility in pure chromium. In these tests, annealed chromium was subjected to 200,000 psi pressure. The results are not conclusive, showing one pressurized specimen manifesting 5.5% elongation, and one pressurized and one unpressurized specimen manifesting 0% elongation. Fracture strain was 30,000 - 40,000 psi for the pressurized specimens, and about 48,000 psi for the unpressurized.

The PTCA experiments were intended for comparison with the above results on pure chromium. All specimens

were of A.S.M. type, 1/4" diameter in a 1" gage length, overall specimen length about 3". A control specimen which was not subjected to any pressurization manifested an upper yield point of 32,600 psi, a lower yield point of 31,000 psi, an ultimate tensile strength of 40,000 psi, and a per cent reduction in area of 3.5. The fracture was brittle occurring at the points of contact of the extensometer with the specimen. A second specimen was pressurized to 150,000 psi in gasoline, held at pressure for one minute and returned to ambient conditions. This specimen manifested a 0.2% yield strength of 31,400 psi, an ultimate tensile strength of 46,000 psi, and a per cent reduction in area of 3.1. Fracture again was brittle occurring at the points of contact of the extensometer with the specimen.

A third specimen was encased in a cylindrical jacket, about 0.5" outside diameter, and the entire assembly was subjected to a pressure of 450,000 psi using a hydraulic oil as the pressure transmitting fluid. After the pressure was reduced to atmospheric, the specimen was essentially unchanged in dimension and shape, and was found to possess 0.2% yield strength of 44,000 psi, and ultimate tensile strength of 47,700 psi, and a per cent reduction in area of 4.4. Fracture was brittle, occurring at points of contact

of the extensometer with the specimen.

Both pressurization treatments eliminated the upper yield point. The simple pressurization to 150,000 psi was not found to appreciably affect any of the physical properties of the material. The pressurization with a compressible jacket was found to increase the yield strength by about one-third, a result similar to that previously obtained (unpublished) on nickel, and on iron as in reference 4.

The tensile tests of pressurized and unpressurized specimens were all conducted at a crosshead speed of .01" per minute, equivalent to a strain rate of .01" per minute in the 1" gage length assuming no major deformation in threads or tensile fixtures. The Australian data (unavailable at the time of conducting these tests) indicate that crosshead speeds of .005" per minute and higher may be responsible for brittle behavior in pressurized specimens.

The conclusions that may be drawn from these tests are consequently restricted to the following:

1. Chrome-30, pressurized to 150,000 psi and higher, does not manifest ductile behavior in a tensile test conducted at a strain rate of .01 per minute at ambient temperature.
2. Pressurization in a compressible jacket to 450,000 psi can increase the 0.2% yield strength

of Chrome-30 by about one-third, in tensile tests at a strain rate of .01" per minute at ambient temperature.

Chrome-30 (Chromium-MgO Composite) - Tensile Tests

A tensile specimen of 1/2" gage length (overall length .9995" and of a .160 nominal diameter (.1593" actual diameter) was prepared with a polished finish although light tool marks were still evident. This specimen manifested a yield stress of 29,000 psi and UTS of 67,500 psi in a test with fracture at 167,000 psi (environmental). The specimen manifested 29.5% elongation and 50% reduction in area. The fracture did not show localized necking, but instead the gage diameter tapered more or less uniformly down to the cross sectional area of failure. Also, the cross sectional area at fracture was somewhat elliptical instead of being round. It is possible that this anisotropic behavior may be a result of unequal densification in different directions across the cross section during the manufacture of the material.

The loading of a tensile specimen under pressure is incremental in that straining of the specimen is halted whenever data is taken. It is consequently not possible to express the strain rate as a continuous quantity. It may be noted, however, that 61 data points were taken

during a test which required about six hours. It is estimated that the actual strain rate during load increments was in the order of .004" per minute to .015" per minute.

These results tend to indicate that the metal-ceramic composite is behaving as is usual with metals in general under pressure, namely, becoming more ductile as environmental pressure is increased.

Mar-Aging Steel - Fluid Extrusion

The annealed mar-aging steel previously reported manifested the following properties in tensile tests conducted on the fluid-to-fluid-extruded material.

TABLE 1

<u>% Reduction</u>	<u>.2% Y.S., ksi</u>	<u>TS, ksi</u>	<u>% El. (4D)</u>	<u>% Red. Area</u>
0	122.0	155.5	20.3	71.5
48	167.1	170.9	13.8	68.0
57	166.7	171.3	13.2	67.0

The hardened mar-aging steel fluid-to-fluid extruded material tensile test results are shown in Table 2. Unfortunately, one sample was ruined in the machining of a tensile specimen from it.

TABLE 2

<u>% Reduction</u>	<u>.2% Y.S., ksi</u>	<u>TS, ksi</u>	<u>% El. (4D)</u>	<u>% Red. Area</u>
0	273.2	277.0	13.0	55.5
28	278.5	282.9	10.0	55.0
57	Ruined in machining			

Tensile samples were machined from the fluid-to-fluid-extruded samples with a reduced section of .100" diameter and a gage length of .500". Tensile tests on all specimens were conducted by the Allegheny-Ludlum Steel Corporation who kindly supplied all samples for this work.

It may be concluded that the reductions employed on the mar-aging steel samples were sufficiently great to increase strength, to reduce per cent elongation, and to not appreciably affect per cent reduction in area. It is possible that the hardened mar-aging steel is the highest-strength material in the world yet worked by fluid-to-fluid extrusion without fracture.

NASA Nickel Base TAZ-8 Alloy

A billet of TAZ-8 alloy was fluid-to-fluid extruded with 16% reduction. There were some initial transverse cracks which changed to a relatively uncracked section

toward the end of the extrusion. The aft end of the extrusion showed definite anisotropy due to varying orientations of relatively large grains contained therein. Finish was high.

It is believed that sound extrusions can be made of this material although there will probably continue to be a grain-size effect due to the relatively large size of grains (some about as large as the die opening).

There appears to be relatively high-billet-die friction in these runs. It was found that a coating of indium metal provided good lubrication.

Magnesium Oxide

A tensile test was attempted of magnesium oxide single crystal, diamond-ground. During pressurization of the pressure chamber containing the specimen, a noise was heard. The run was aborted, and the single crystal was found to be cracked, from no discernible cause. A tensile test was made of polycrystalline magnesium oxide having a diamond-ground finish. The pressure at fracture was 290,000 psi. The yield strength was 40,000 psi, ultimate tensile strength 50,000 psi, and per cent elongation was 6%. There was no discernible reduction in area. Since there was no reduction in area in the tensile test, even at the high

environmental pressure used, it was not felt worthwhile to attempt any extrusion tests.

Tungsten-Tantalum Alloy

There was supplied by the sponsor an arc-cast tungsten - 4.5% tantalum ingot. There were machined from this ingot tensile specimens as well as extrusion specimens. A tensile test was conducted on tungsten-tantalum alloy at a pressure of 311,000 psi. The material manifested a yield strength of 120,000 psi, an ultimate tensile strength of 150,000 psi, 4 1/4% elongation, and 10% reduction in area. It is believed that these results are typical not only of the material in the specific condition obtained, but also of the orientation. If the cylindrical ingot is designated in direction as axial, radial and tangential, the specimen employed in this test would be tangential. The fracture was brittle and normal to the axis of the tensile specimen. This material exhibited an extraordinarily large tendency to crack during grinding. The cracks were sufficiently difficult to detect that all specimens were etched by a mixture of hydrochloric and nitric acid much as would be done with tungsten alone.

The resultant rough-surface finish posed the problem as to whether the billet could be sealed in the die during

a fluid-to-fluid extrusion process without leakage between the asperities on the surface. A preliminary and crack-free partial extrusion was made with 7% reduction that effectively demonstrated that this rough-surface material could be handled routinely in the fluid-to-fluid extrusion process. The figure shows the partial extrusion on this billet. The etched nature of the surface is visible.



FUTURE WORK

Future work will be devoted to a continuation of the studies reported herein.

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